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DOCUMENT-IDENTIFIER: US 6015506 A
TITLE: Composition and method for polishing rigid disks

Brief Summary Text (3):
This invention concerns a method for polishing rigid disks (which are used for computer hard drives) using an aqueous dispersion of fine abrasive particles. The aqueous dispersion may be incorporated into a chemical mechanical polishing slurry including at least one oxidizer and an optional catalyst. More particularly the method of this invention is especially adapted for polishing electroless nickel deposited rigid disks.

Brief Summary Text (5):
Great strides are being made in the miniaturization of electronic components for the computer and electronics industries. Miniaturization has created component quality concerns, many of which are resolved by the precise polishing of computer and electronic substrate materials for magnetic disks and semi-conductors. As a result, identifying methods and compositions that can produce an essentially defect free surface has become crucial in the manufacture of computer and electronic substrates.

Brief Summary Text (6):
The driving force for miniaturization in rigid disks is similar to that in the semiconductor industry. Customers are demanding continually increasing storage capacity in rigid disks. At the same time the design rules used by computer manufacturers call for smaller hard drives. The only solution available to rigid disk manufacturers is to increase the storage density of the magnetic media. The recent development of magneto-resistive head technology allows heads to "float" at less that 50 nm above the disc surface at rotation rates in excess of 5000 rpm. Therefore a quantum leap is needed in surface finish quality of rigid disks as measured by such parameters as rms roughness and defectivity in order to enhance the signal to noise ratio of finished rigid disks.

Brief Summary Text (7):
Dispersions and chemical mechanical polishing (CMP) slurries have been developed for use in conjunction with semi-conductor device manufacture. However, few of the commercially available dispersions or CMP slurries have been evaluated in rigid disk polishing applications. For example, U.S. Pat. No. 4,475,981 discloses a composition for polishing the metal surface of nickel plated blanks for rigid memory disks with a composition including ceric oxide or aluminum oxide powder, a water soluble chlorine-containing mild oxidizing agent and an aqueous suspension of colloidal alumina oxide or ceria oxide. U.S. Pat. Nos. 4,696,697 and 4,769,046, each disclose methods for polishing memory disks using an abrasive composition including alpha-aluminum oxide and a polishing accelerator such as nickel sulfate. The aluminum oxide polishing agent preferably has a minum particle size of 0.7-4 .mu.m and a maximum particle size of 20 .mu.m or less. U.S. Pat. Nos. 4,915,710 and 4,929,257 each disclose abrasive compositions suitable for polishing aluminum based substrates for magnetic recording disks. The composition disclosed includes an alumina abrasive, a polishing accelerator such as gluconic or lactic acid and colloidal alumina. In addition, U.S. Pat. No. 5,527,423 discloses an abrasive composition that is particularly useful in the method of this invention. Likewise, U.S. patent application Ser. No. 08/753,482, incorporated herein by reference,

discloses a chemical mechanical abrasive composition including an oxidizer and a catalyst that is useful in the method of this invention.

Brief Summary Text (8):
Methods for polishing rigid disks are disclosed in U.S. Pat. Nos. 4,769,046, 5,084,071, and 5,441,788. U.S. Pat. No. 4,769,046 discloses a method for polishing a layer of nickel plated on a rigid disk using a composition comprising aluminum oxide and a polishing accelerator such as nickel nitrate, aluminum nitrate, or mixtures thereof. U.S. Pat. No. 5,084,071 discloses a method of chemical mechanical polishing and electronic component using a composition including abrasive particles that are not alumina, a transition metal chelated salt, a solvent for the salt, and a small but effective amount of alumina. Finally, U.S. Pat. No. 5,441,788 discloses a method for manufacturing a nickel phosphor recording disk including polishing the NiP substrate to a surface roughness to no less than 2.0 nm RMS.

Brief Summary Text (9):
The commercially available rigid disk polishing slurries are unable to meet the new rigid disk surface finish parameters. Furthermore, compositions that are presently known to be capable of polishing rigid disks are unable to polish rigid disks at a high rate and, at the same time, produce an acceptable surface finish. Therefore, there remains a need for dispersions, and chemical mechanical polishing slurries that are capable of polishing rigid or hard disks at high rates with low defectivity, while providng a smooth surface finish.

Brief Summary Text (11):
The present invention is directed to a method for polishing rigid disks using a fine particulate dispersion alone, or incorporated into a chemical mechanical polishing slurry. The method of this invention is able to remove nickel phosphide from an aluminum alloy-based rigid disk at high rates while producing a rigid disk with an essentially defect free surface.

Brief Summary Text (14):
In one embodiment, this invention is a method for polishing a rigid disk. The method uses a dispersion comprising a metal oxide abrasive having a surface area ranging from about 5 m.sup.2 /g to about 430 m.sup.2 /g, a particle size distribution less than about 1.0 micron, and a mean particle diameter less than about 0.4 microns and a pH of from about 2.0 to about 7.0. The dispersion is applied to at least one surface of the rigid disk and a polishing pad is moved into contact with the surface of the rigid disk. The polishing pad is moved in relation to the rigid disk to give a polished rigid disk having an rms roughness of less than 1.4 nm.

Brief Summary Text (15):
In another embodiment, this invention is a method for polishing a rigid disk having a first side and a second side. The method uses a chemical mechanical polishing slurry comprising a fumed alumina abrasive having a surface area ranging from about 5 m.sup.2 /g to about 430 m.sup.2 /g, a particle size distribution less than about 1.0 micron, a mean particle diameter less than about 0.4 microns and a pH of about 2.0 to about 5.0, an iron catalyst, and from about 0.1 to about 10.0 weight percent of an oxidizing agent selected from the group consisting of hydrogen peroxide and monopersulfate wherein when the oxidizing agent is hydrogen peroxide, then the slurry includes from about 0.01 to about 0.20 weight percent ferric nitrate and when the oxidizing agent is monopersulfate, then the slurry includes from about 0.1 to about 0.5 weight percent ferric nitrate catalyst. The chemical mechanical polishing slurry is applied to the first surface and the second surface of the rigid disk. A first pad is moved into contact with the first surface and a second pad is moved into contact with the second surface, and the first pad and the second pad are moved in relation to the rigid disk to give a polished rigid disk having an rms roughness of less than 1.4 nm.

Brief Summary Text (17):
In still another embodiment, this invention is a dispersion comprising a metal oxide abrasive having a surface area ranging from about 5 m.sup.2 /g to about 430 m.sup.2 /g, a particle size distribution less than about 1.0 micron, a mean particle diameter less than about 0.4 microns, and a pH of from about 2.0 to about 7.0. More particularly, this invention is a chemical mechanical polishing slurry incorporating

the dispersion and one or more ingredients selected from an oxidizing agent, a metal catalyst, and additives.

Brief Summary Text (19):
The present invention relates to abrasive dispersion compositions of matter useful for polishing rigid disks comprising a fine dispersion having distinct particle characteristics. The present invention also relates to a method for polishing rigid disks using an abrasive dispersion alone or combined with specific additives including at least one oxidizer and, optionally, at least one catalyst to give a chemical mechanical polishing composition (CMP slurry) that promotes a chemical reaction between the oxidizer and the rigid disk metal layer. In particular, both the dispersion and the chemical mechanical polishing slurry are capable of polishing rigid disks at good rates to give polished rigid disks having excellent RMS roughness ranging from about less than 1.4 nm at polishing rates greater than about 1.5 .mu.inch/min.

Brief Summary Text (20):
Before describing the details of the various preferred embodiments of this invention, some of the terms that are used herein will be defined. The term chemical mechanical polishing slurry as used herein, ("CMP slurry"), refers to the combination of at least one abrasive dispersion and at least one oxidizer.

Brief Summary Text (21):
The term "rigid disk" refers to rigid disks and hard disks, such as an aluminum disk or nickel phosphor (NiP) plated aluminum disk upon which a magnetic media for computer memories will be coated.

Brief Summary Text (22):
One aspect of this invention is method for polishing rigid disks using a dispersion of an abrasive with specific particle characteristics that has heretofore never been appreciated as being useful for polishing rigid disks. An abrasive useful in the dispersion of this invention is described in U.S. Pat. No. 5,527,423 which is incorporated herein by reference. The abrasive described in the '423 patent has well defined particle properties. The preferred metal oxide abrasive will have a surface area, as calculated from the method of S. Brunauer, P. H. Emmet, and I. Teller, J. Am. Chemical Society, Volume 60, Page 309 (1938) and commonly referred to as BET, ranging from about 5 m.sup.2 /g to about 430 m.sup.2 /g, more preferably from about 20 m.sup.2 /g to about 250 m.sup.2 /g, and most preferably from about 30 m.sup.2 /g to about 100 m.sup.2 /g when the abrasive is fumed alumina and about 40 m.sup.2 /g to about 200 m.sup.2 /g when the abrasive is fumed silica. Due to stringent purity requirements in the IC industry the preferred metal oxide should be of a high purity. High purity means that the total impurity content, from sources such as raw material impurities and trace processing contaminants, is typically less than 1% and preferably less than 0.01% (i.e., 100 ppm).

Brief Summary Text (23):
The metal oxide abrasive useful in the dispersion of this invention may consist of metal oxide aggregates or individual single sphere particles. The term "particle" as it is used herein refers to both aggregates of more than one primary particle and to single particles. The metal oxide particles useful in the methods and compositions of this invention will have a size distribution less than about 1.0 micron, a mean individual or aggregate diameter less than about 0.4 micron and a force sufficient to repel and overcome the van der Waals forces between the abrasive aggregates or individuals particles themselves. Such metal oxide abrasives have been found to be effective in minimizing or avoiding scratching, pit marks, divots and other surface imperfections during polishing of rigid disks. The particle size distribution in the present invention may be determined utilizing known techniques such as transmission electron microscopy (TEM). The mean particle diameter and mean refers to the average equivalent spherical diameter when using TEM image analysis, i.e., based on the cross-sectional area of the particle. By force is meant that either the surface potential or the hydration force of the metal oxide particles must be sufficient to repel and overcome the van der Waals attractive forces between the particles.

Brief Summary Text (25):
The metal oxide abrasive may be selected from the group including alumina, titania,

zirconia, germania, silica, ceria mixtures thereof and chemical admixtures thereof. The term "chemical admixture" refers to particles including atomically mixed or coated metal oxide abrasive mixtures. The dispersion useful in the method of this invention preferably includes from about 0.5 to about 55.0 weight percent or more of an abrasive. It is more preferred, however, that the dispersion and/or the CMP slurry of this invention is diluted at the point of use with deionized water or any other acceptable diluent to produce a dispersion with less than 12 wt %, preferably less than about 6.0 wt % abrasive when the abrasive is alumina, less than about 10.0 wt % abrasive when the abrasive is silica, and most preferably from about 3.0 to about 6.0 weight percent abrasive. The term "point of use" refers to the location where the dispersion is used which, in the case of a rigid disk, would be the surface of a rigid disk undergoing polishing.

Brief Summary Text (27):
For example, the production of fumed metal oxides is a well-known process which involves the hydrolysis of suitable feedstock vapor (such as aluminum chloride for an alumina abrasive) in a flame of hydrogen and oxygen. Molten particles of roughly spherical shapes are formed in the combustion process, the diameters of which are varied through process parameters. These molten spheres of alumina or similar oxide, typically referred to as primary particles, fuse with one another by undergoing collisions at their contact points to form branched, three dimensional chain-like aggregates. The force necessary to break aggregates is considerable and often considered irreversible. During cooling and collecting, the aggregates undergo further collision that may result in some mechanical entanglement to form agglomerates. Agglomerates are thought to be loosely held together by van der Waals forces and can be reversed, i.e., de-agglomerated, by proper dispersion in a suitable media.

Brief Summary Text (29):
The manufacture of gel based materials, e.g. aerogels, xerogels, hydrogels and other gels is well known to those skilled in the art and may be accomplished utilizing conventional techniques, for example, U.S. Pat. No. 3,652,214 to Aboutboul, et al., U.S. Pat. No. 2,188,007 to Kistler, and as disclosed in the article by Heley, et al., entitled "Fine Low Density Silica Powders Prepared by Supercritical Drying of Gels Derived From Silicon Tetrachloride," Journal of Non-Crystalline Solids, 186, 30-36 (1955).

Brief Summary Text (30):
The metal oxide abrasives produced by the processes described above can be further processed by pulverization and crushing processes to give metal oxide abrasives that are useful in the methods and compositions of this invention. Useful pulverization and crushing processes include milling or grinding using conventional manufacturing techniques such as jet-milling, ball milling, bead milling, and other milling and pulverization techniques and process know to one skilled in the art.

Brief Summary Text (31):
Preferably, the metal oxide is a fumed or precipitated abrasive and, more preferably it is a fumed abrasive such as fumed silica or fumed alumina. Fumed alumina may be comprised of amorphous alumina, high temperature crystalline phases of alumina consisting of gamma, theta, delta, and alpha alumina, and low temperature phases of alumina consisting of all non-high temperature crystalline alumina phases.

Brief Summary Text (32):
It is preferred that the fumed alumina used in the compositions of this invention has a crystalinity of at least 40 weight percent. It is more preferred that the fumed alumina used in the present invention is greater than 90 weight percent crystalline, and that the fumed alumina consist of at least 80 weigh percent high temperature crystalline phases of alumina.

Brief Summary Text (34):
Another aspect of this invention is a method for polishing rigid disks that uses a chemical mechanical composition that has heretofore never been appreciated as being useful for polishing rigid disks. A chemical mechanical composition useful for polishing rigid disks according to this invention includes an oxidizing agent and an optional catalyst. This chemical mechanical composition is useful when mixed with at

least one abrasive to give a chemical mechanical polishing slurry that is useful for polishing rigid disks. The oxidizing agent--catalyst combinations disclosed herein are useful when incorporated into a CMP slurry or when used alone as a chemical mechanical composition in conjunction with an abrasive pad to polish metals and metal based components of rigid disks. Alternatively, the chemical mechanical polishing slurry of this invention may include a metal oxide abrasive and an oxidizing agent without a catalyst.

Brief Summary Text (35):
The chemical mechanical composition and slurry useful in this invention is disclosed in U.S. patent application Ser. No. 08/753,482 which is incorporated herein by reference. The useful chemical mechanical compositions includes at least one oxidizing agent. Suitable oxidizing agents include oxidizing metal salts, oxidizing metal complexes, nonmetallic oxidizing acids such as peracetic and periodic acids, iron salts such as nitrates, sulfates, EDTA, citrates, potassium ferricyanide, hydrogen peroxide, potassium dichromate, potassium iodate, potassium bromate, vanadium trioxide and the like, aluminum salts, sodium salts, potassium salts, ammonium salts, quaternary ammonium salts, phosphonium salts, or other cationic salts of peroxides, chlorates, perchlorates, nitrates, permanganates, persulfates and mixtures thereof.

Brief Summary Text (36):
When a catalyst is used in the composition and method of this invention, it is preferred that the oxidizing agent chosen have an electrochemical potential greater than the electrochemical potential necessary to oxidize the catalyst. For example an oxidizing agent having a potential of greater than 0.771 volts versus normal hydrogen electrode is necessary when a hexa aqua iron catalyst is oxidized from Fe(II) to Fe(III). If an aqua copper complex is used, an oxidizing agent having a potential of greater than 0.153 volts versus normal hydrogen electrode is necessary to oxidize Cu(I) to Cu(II). These potentials are for specific complexes only, and may change, as will the useful oxidizers, upon the addition of additives such as ligands (complexing agents) to the compositions of this invention.

Brief Summary Text (39):
The oxidizing agent may be present in a chemical mechanical polishing slurry in an amount ranging from about 0.01 to about 50.0 weight percent and preferably from about 0.01 to about 10.0 weight percent. It is preferred that the oxidizer is present in the slurry in an amount ranging from about 0.1 to about 5.0 weight percent when the oxidizing agent is a monopersulfate, and from about 0.2 to about 10.0 wt % when the oxidizing agent is a triple salt.

Brief Summary Text (40):
The useful chemical mechanical composition, of this invention includes at least one catalyst. The purpose of the catalyst is to transfer electrons from the metal being oxidized to the oxidizer (or analogously to transfer electrochemical current from the oxidizer to the metal). The catalyst or catalysts chosen may be metallic, non-metallic, or a combination thereof and the catalyst must be able to shuffle electrons efficiently and rapidly between the oxidizer and metal substrate surface. Preferably, the catalyst is chosen from metal compounds that have multiple oxidation states, such as but not limited to Ag, Co, Cr, Cu, Fe, Mo, Mn, Nb, Ni, Os, Pd, Ru, Sn, Ti and V. The term "multiple oxidation states" refers to an atom and/or compound that has a valence number that is capable of being augmented as the result of a loss of one or more negative charges in the form of electrons. Most preferred metal catalysts are compounds of Ag, Cu and Fe and mixtures thereof. Especially preferred are iron catalysts such as but not limited to inorganic salts of iron, such as iron (II or III) nitrate, iron (II or III) sulfate, iron (II or III) halides, including fluorides, chlorides, bromides, and iodides, as well as perchlorates, perbromates and periodates, and ferric organic iron (II or III) compounds such as but not limited to acetates, acetylacetonates, citrates, gluconates, oxalates, phthalates, and succinates, and mixtures thereof.

Brief Summary Text (41):
The catalyst may be present in the chemical mechanical polishing composition in an amount ranging from about 0.001 to about 2.0 weight percent. It is preferred that the catalyst will be present in the chemical mechanical polishing composition in an

amount ranging from about 0.005 to about 0.5 weight percent. At this preferred catalyst loading level, i.e., 0.5 weight percent or less, and when a non-metallic oxidizing agent such as hydrogen peroxide, urea hydrogen peroxide or monopersulfate is used, the chemical mechanical polishing composition is essentially metal and "metallic ion free" in comparison to commercially available ferric nitrate based slurries.

Brief Summary Text (42):
The amount of catalyst used may vary depending upon the oxidizing agent used. When the preferred oxidizing agent hydrogen peroxide is used in combination with a preferred catalyst such as ferric nitrate, the catalyst will preferably be present in the composition in an amount ranging from about 0.01 to about 0.20 weight percent (approximately 7 to 280 ppm Fe in solution) and preferrably from about 0.01 to about 0.05 weight percent ferric nitrate. When the preferred oxidizing agent is a triple salt of monopersulfate and a preferred catalyst such as ferric nitrate is used, the catalyst will be present in the composition in an amount ranging from about 0.05 to about 1.0 weight percent (approximately 70 to about 1400 ppm Fe in solution) and preferably from about 0.1 to about 0.5 weight percent.

Brief Summary Text (43):
The concentration ranges of catalyst in the chemical mechanical polishing slurry of this invention are generally reported as a weight percent of the entire compound. The use of high molecular weight metal containing compounds that comprise only a small percentage by weight of catalyst is well within the scope of catalysts in this invention. The term catalyst when used herein also encompasses compounds wherein the catalytic metal comprises less than 10% by weight of the metal in the composition and wherein the metal catalyst concentration in the CMP slurry is from about 2 to about 3000 ppm of the overall slurry weight.

Brief Summary Text (44):
The chemical mechanical composition may be combined with at least one abrasive to produce a CMP slurry. The abrasive may be any metal oxide abrasive. However, the metal oxide abrasive described above and in U.S. Pat. No. 5,527,423, disclosed herein by reference, are preferred.

Brief Summary Text (45):
Other well known polishing slurry additives may be incorporated alone or in combination into the chemical mechanical polishing slurry useful in the process of this invention. A non-inclusive list is inorganic acids, organic acids, surfactants, alkyl ammonium salts or hydroxides, and dispersing agents.

Brief Summary Text (46):
An additive which may be useful with this invention is one which stabilizes the oxidizer in the presence of the metal complex. It is well known that hydrogen peroxide is not very stable in the presence of many metal ions without the use of stabilizers. Useful stabilizers include phosphoric acid, organic acids (e.g., acetic, citric, tartaric, orthophthalic, and EDTA), tin oxides, phosphonate compounds and other ligands which bind to the metal and reduce its reactivity toward hydrogen peroxide decomposition. These additives can be used alone or in combination and significantly decrease the rate at which hydrogen peroxide decomposes, and may also effect tungsten polishing rates.

Brief Summary Text (47):
Metal surfaces such as nickel-plated blanks for rigid memory discs are polished with the above described compositions by subjecting the surface to mechanical rubbing (polishing) in the presence of the composition. The rubbing effects mechanical smoothing or wear of the surface which is aided by the abrasive, and when present, promoted by the components added to the abrasive to give a chemical mechanical polishing slurry which chemically attacks and dissolves the components comprising the disk metal surface. Polishing may thus by achieved solely by a mechanical mechanism, or by a combination of chemical and mechanical mechanisms.

Brief Summary Text (50):
Rigid disk polishing can be accomplished in one or two steps. In the first step, an abrasive dispersion or chemical mechanical polishing slurry is applied to the rigid

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